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# Development of organomineral fertilisers derived from nutrient-enriched biosolids granules: product specification

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***Abstract.*** *A technique was developed to produce organomineral fertilisers (OMF) using biosolids granules coated with urea and potash. Two OMF formulations with the following N:$P_2O_5$:$K_2O$ compositions: 10:4:4 ($OMF_{10}$) and 15:4:4 ($OMF_{15}$) were developed for application in grassland and arable crops. Routine fertiliser analyses were conducted on OMF and biosolids samples, and compared with a sample of urea to determine key physical and chemical properties of the materials which affect handling and spreading, soil behaviour and fertiliser value. Bulk and particle densities were in the range of 608 to 618 kg $m^{-3}$, and 1297 to 1357 kg $m^{-3}$ respectively. Compression tests showed that OMF particles undergo deformation followed by multiple failures without disintegration of the granules when vertical load was applied. This behaviour was attributed to the moisture content (≈15% w $w^{-1}$) and the organic nature of the material. Static particle strength was between 1.14 and 6.31 N $mm^{-2}$ depending on the particle diameter. The use of a model for fertiliser particle distribution studies showed that OMF granules should be between 1.10 and 5.50 mm in diameter with about 80% of the particles in the range of 2.25 to 4.40 mm to enable application at 18 m tramline spacing. This research is novel as it utilises technology to improve fertiliser value of biosolids to increase usage by farmers, reduce disposal costs and deliver the range of environmental benefits associated with recycling.*

***Keywords.*** *Organomineral fertilisers (OMF), physical and chemical properties, biosolids granules, recycling.*

# Introduction

## *The need for recycling*

Estimates (DEFRA, 2007a) indicated that the production of sewage sludge (biosolids) in England and Wales is approximately 1.6 million tonnes per year. This amount is set to increase due to the growth of the population and the continuous adoption of improved technologies which are introduced to comply with the required standards for the treatment of effluents. More than 10 million tonnes (dry solids) of sewage sludge are produced annually in the EU (Laturnus et al., 2007). The quantity of sewage sludge recycled to agriculture varies considerably between EU member states. Ireland recycles approximately 90%, while Finland, Slovenia, Sweden, Holland, Greece and Belgium recycle very low quantities or no sewage sludge to agriculture (Smith, 2008). Conventional routes for disposal of sewage sludge include landfill and incineration which are considered to be unsustainable and therefore being progressively restricted. In this respect, the EU Landfill Directive 99/31/EC (CEC, 1999) requires reduction of 35% of biodegradable waste by 2020 of that generated in 1995 while the UK Government is committed to cut back current levels of $CO_2$ emission by 20% (DEFRA, 2007b).

Expanding incineration infrastructure to deal with increased sewage production can be cumbersome due to difficulties arising from the approval of planning permissions and also because of strong public opposition. Recycling of biosolids to agricultural land is relatively less expensive compared with other disposal practices i.e. 30%-40% compared with incineration and landfill per tonne of raw sludge (dry solids) (Antille, 2011), and it is regarded as the best practicable environmental option in most circumstances (Edge, 1999). The practice of recycling supports the waste management hierarchy (DEFRA, 2007a) and the principles of sustainable intensification of agriculture (Royal Society of London, 2009). It is envisaged that the use of organic-based fertiliser materials will increase in the near future (Paré et al., 2009) as a result of technological developments that may enable the production of high quality products, improvements in application techniques for field spreading and specialist equipment, and cost advantages compared with mineral fertilisers.

The expected increase in the global population with the associated rise in demand for food and energy will increase the reliance on fertiliser inputs (Dawson and Hilton, 2011). The demand for N, P and K is forecasted to increase at an average rate of about 2.5% per year to 2020 (Heffer and Prud'homme, 2010). Since phosphate reserves are limited (Herring and Fantel, 1993), there is a need to ensure that phosphorus is recycled to a large extent so that the rate of depletion of phosphate rock is reduced (Weikard and Seyhan, 2009; Dawson and Hilton, 2011). Dawson and Hilton (2011) argued that legislation concerning management of phosphorus in agriculture appears entirely linked to its potential to disturb natural ecosystems with apparently no regulations yet requiring the efficient use and reuse of a finite resource.

In recent years, there has been a progressive increase in the price of mineral fertilisers (Nix, 2012). This trend is likely to continue driven by the projected increase in fertiliser demand and the cost of energy with special regards to mineral nitrogen fertilisers (Heffer and Prud'homme, 2010). For example, the price of straight nitrogen fertilisers increased from approximately GBP0.25 to 0.60 per kg of N between 1994 and 2010 but it reached GBP1.05 per kg of N in 2008 (Nix, 2012). Enhanced organic-based fertiliser materials can provide an opportunity to improve crop profit margins by means of reduced input costs of fertilisers while delivering some of the environmental benefits associated with recycling.

Development of new fertiliser products requires determining key properties of the materials that affect storage and spreading, soil behaviour and agronomic efficiency.

The information available describing physical characteristics of organomineral fertiliser products is limited (e.g. Allaire and Parent, 2003a-b; Paré et al., 2009) which responds to the lack of legal requirements for their declaration on the label (Allaire and Parent, 2004). Whilst the EC has made attempts to define a common regulation for organic-based fertiliser materials, the proposal for a Directive made in the 1990's had no success at country level and therefore could not be taken forward or implemented (Graziano et al., 2008).

Conversely, the physical properties of mineral fertilisers have been studied in detail e.g. Hofstee and Huisman, 1990; Miller, 1996; Agnew and Leonard, 2003. Miller (1996) indicated that fertiliser materials which have moderately high crushing strength can resist handling, storage and spreading without significant shattering, dust-formation or caking. Density properties are related to the volume required for storage and transport, and are required to calibrate fertiliser spreading equipment (Miller, 1996; Paré et al., 2009). Particle size and size distribution affect uniformity of distribution during field application (Hofstee, 1993) and it is well documented that uneven spreading of fertilisers can increase nutrient losses to the environment, reduce fertiliser use efficiency and crop profit margins (Dilz and van Brakel, 1985; England and Audsley, 1987).

## *Organomineral fertilisers*

The literature abounds with reported experiences in the use of organomineral fertilisers applied to a variety of crops with satisfactory agronomic performances. Organomineral fertiliser is defined as a fertiliser obtained by blending, chemical reaction, granulation or dissolution in water of inorganic fertilisers having a declarable content of one or more primary nutrients with organic fertilisers or soil improver (Anon., 2012). These products have been derived from a range of different organic and inorganic sources e.g. Richards et al. (1993), Efanov et al. (2001), Makinde (2007), WIPO (2009), Paré et al. (2010), Rady (2012). Zebarth et al. (2005) proposed the use of organic-based fertiliser materials to mitigate potential environmental effects associated with the use of mineral fertilisers alone which usually release their nutrients more rapidly following soil application. In such materials, the organic fraction protects the inorganic components by means of binding and absorption thereby slowing the rate of release of plant nutrients (FAO, 2007). Tejada et al. (2005), however, suggested that the existence of a time-lag between soil application and nutrients uptake by the crop can increase the risk of nutrient losses to the environment.

In the UK, some wastewater companies commercialise treated biosolids pellets available in one tonne bags for agricultural use but they are produced without the addition of mineral fertilisers (Spence, 2010). The present article focuses on the study of the physical and chemical characteristics of organomineral fertilisers which are produced by coating biosolids with urea and potash. A patent application (US7504035-B2) was assigned to United Utilities Group PLC (2009) for the treatment of putrescrible cakes for the reduction of *Escherichia coli* and odour both of which are required in this product for safe spreading on agricultural land. The use of biosolids-derived organomineral fertilisers can reduce the cost of spreading by about 30% compared with biosolids when these are applied at the optimum N rate in winter cereal crops (Antille, 2011). Cost savings and differences in working rates become greater when compared with liquid sludges requiring injection (Antille, 2011).

The main aim of this development was to design a product that could satisfy the requirements of modern agricultural practices, maximise the use of biosolids in crop production; in particular, in areas close to production, and ultimately reduce the reliance on mineral fertilisers. In the UK, the GB Fertiliser Regulations are currently considering renewable sources of phosphorus but discussions are still in their infancy. The use of biosolids-based organomineral fertilisers addresses an important issue of nutrient cycling between urban and agricultural ecosystems.

It represents a technological advancement compared with the ways that sewage sludge has been traditionally recycled in agriculture and it appears to be in line with the current environmental and regulatory frameworks. The specific objective of this study was to characterise chemical and physical properties required to meet specifications for organomineral fertilisers (OMF) derived from nutrient-enriched biosolids granules for application in grassland and arable crops.

## Materials and Methods

### *Description of the products*

The organomineral fertilisers are produced by drying digested sewage sludge cake (25% dry solids) at 80°C in a tumble dryer which produces granules of varying diameter. As a result, the dry solids content of the sludge is increased to about 80% to 85%. Subsequently, granules are coated, by means of spraying, with melted urea and ground potash to raise the concentration of nitrogen and potassium respectively. The final product is a compound NPK organomineral fertiliser as shown in Figure 1. The core of the granules (biosolids) releases nutrients more slowly compared with the mineral fraction (urea and potash) (Antille et al., 2012).

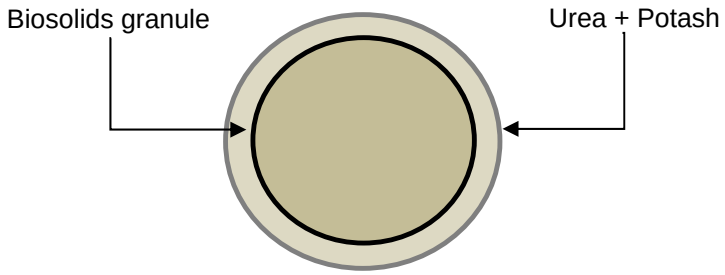


Figure 1: A schematic drawing of an organomineral fertiliser (OMF) particle.

The land spreading of sewage sludge (biosolids) is regulated by the Sludge (Use in Agriculture) Regulations 1989 S.I. No.: 1263 which implements the provisions of the EU Sewage Sludge Directive 86/278/EEC (CEC, 1986) in Great Britain regarding the protection of the environment. The treatment of sewage sludge (biosolids) is a requirement prior to land application (ADAS, 2001). A process known as high rate enzyme hydrolysis is employed to control the pathogens load in biosolids (Le et al., 2006). This process takes place during the anaerobic digestion and it provides up to 99.9999% pathogens destruction (Le et al., 2006) enabling compliance with the Safe Sludge Matrix (ADAS, 2001).

### *Product formulation*

The organomineral fertiliser (OMF) products were suggested for broadcast application in grassland as well as arable cropping. Phosphorus fertilisation in excess of the crop requirements may be avoided in soils that have satisfactory soil P Indexes (DEFRA, 2010). Research (Skinner and Todd, 1998) has shown that about 20% of the soils in Northern England had soil P Index 3 or greater which agrees with more recent information supplied by wastewater operators in the NW region of England (United Utilities Group PLC, 2007). High soil P status imposes a restriction to biosolids recycling and it compromises meeting recycling targets by wastewater companies. It also increases the cost of disposal as biosolids require transportation farther away from wastewater treatment works.

Chemical analyses conducted on samples of biosolids granules reported an average content of 3% N (standard deviation =0.81), 4.35% $P_2O_5$ (standard deviation =1.07) and 0.15% $K_2O$ (standard deviation =0.062). The relatively low content of nitrogen compared to phosphorus results in low N:$P_2O_5$ ratios which are often less than 1. This can lead to a progressive build-up in soil P levels if biosolids are regularly applied based on crop nitrogen requirements. Soil P levels above the target Index for the soil and crop system can have undesirable effects if phosphorus is subsequently lost to the environment (Alvarez-Corbelas et al., 2009). This requires increasing existing N:$P_2O_5$ ratio in the base biosolids to: (1) ensure that both nutrients were supplied to the crop in sufficient quantities for a grain yield of 8 t $ha^{-1}$ considered to be typical of winter wheat crops in England (DEFRA, 2010), and (2) reduce the risk of increasing soil P Index when the fertilisation strategy considered routine application of OMF.

Narrow N:$P_2O_5$ ratios in the product would be suitable for lower P Index soils (e.g. less than 3) allowing for corrections to be made overtime towards the target Index (DEFRA, 2010). Wider N:$P_2O_5$ ratios suit situations where soil P Index is higher but where the overall fertility status of the soil is to be maintained. This requires that phosphorus fertilisation does not exceed but instead replenishes P off-take by the crop. Based on this criterion, the content of phosphorus in the OMF remained equivalent to that of the base biosolids granules. Nitrogen concentrations in the OMF above the suggested 15% are technically possible but this has some drawbacks. Firstly, it increases the manufacturing costs due to higher input cost of urea-N. Secondly, the production of more concentrated nitrogen products can reduce the total amount of biosolids recycled through agriculture. Equally, if the nitrogen concentration in the product was increased, the land-bank for recycling would need to be increased accordingly to meet the same disposal targets compared with biosolids. Estimates (Antille, 2011) suggested that the required land-bank for $OMF_{15}$ would need to be approximately 1.5 times larger than that of $OMF_{10}$ assuming OMF-N was applied at a standard rate of 200 kg $ha^{-1}$ and that the price of nitrogen was equivalent to that of urea-N. For winter wheat crops in soils with P index below 3, the recommended phosphorus application rates (grain yield: 8 t $ha^{-1}$) are between 60 and 85 kg $ha^{-1}$ of $P_2O_5$ (DEFRA, 2010). Supplying these rates of phosphorus with biosolids would require between 1400 and 1950 kg of biosolids per hectare resulting in nitrogen application rates in the range of 140 to 160 kg $ha^{-1}$ based on the nutrients concentrations of biosolids mentioned earlier. For a standard rate of 200 kg $ha^{-1}$ of N (DEFRA, 2010), the concentration of nitrogen in the biosolids, in percentage terms, should be increased from 3% to about 10% and 15% respectively. For potassium, the application of the same amount of biosolids would supply approximately 45 to 65 kg $ha^{-1}$ of $K_2O$ less than the recommended quantities for soils with K Index below 3. Hence, the concentration of potash in biosolids would need to be increased from 0.15% to about 3.5%. From this simple analysis, the formulations of the two OMF products were inferred and the final compositions were approximated to N:$P_2O_5$:$K_2O$ ratios: 15:4:4 ($OMF_{15}$) and 10:4:4 ($OMF_{10}$).

The application of these products is not restricted to soils with P Indexes below 3; however, crop requirements for P should not be exceeded in soils which are above the target soil P Index. This criterion for P fertilisation is supported by the basic principle for phosphorus and potassium management indicated by Dawson (2011) which is to ensure the maintenance of sufficient nutrient reserves in the soil. In soils with adequate nutrient supply, the application of phosphorus and potassium is not to provide these nutrients for the crop being grown but to replace their removal from the soil (Dawson, 2011; Johnston et al., 2001a-b; Johnston, 2005). The narrower N:$P_2O_5$ ratio of $OMF_{10}$ compared with $OMF_{15}$ makes it more suitable for situations with relatively lower soil P Index and vice-versa. Mixtures of these materials with other compatible fertiliser sources (AFCOME, 1995) can be used to match specific soil-crop requirements. The coating technology used to supplement biosolids with mineral fertilisers enables overcoming the expected variability in the chemical composition of sludges (Sommers, 1997) to provide a consistent product.

### *Chemical composition*

Analyses were conducted in compliance with the specifications outlined in The Fertilisers (Sampling and Analysis) Regulations 1991 S.I. No.: 973. Total N was determined based on Dumas (1831). Total P and water soluble P (% $P_2O_5$, w $w^{-1}$), total K (% $K_2O$, w $w^{-1}$) and heavy metals: total cadmium (Cd, mg $kg^{-1}$), total copper (Cu, mg $kg^{-1}$) and total zinc (Zn, mg $kg^{-1}$) were based on MAFF (1986) and BS 7755-3.13 (1998), and determined using Inductively Couple Plasma Emission Spectroscopy (ICPES). Cd was determined due to risk of toxicity at low concentrations and potential transfer to the food chain (Johnston and Jones, 1995). Cu and Zn are essential elements but can be toxic to plants and microorganisms above certain levels therefore maximum permissible levels in soil must be observed (MAFF, 1998). Soluble P (MAFF, 1986) was determined to provide an indication of the fraction readily available for plant uptake. The results reported for the measured chemical properties correspond to the mean of 12 samples (n=12) except for heavy metals and water soluble P (n=3).

### *Physical properties*

Particle size analysis was based on BS EN 1235 (1995). Fertiliser samples of 500 g were passed through a series of sieves with apertures in the range of 0.60 to 37 mm. The sieves were placed on a shaker for three minutes and the fertiliser retained was weighed and expressed as percentage of the total weight of the sample. From the particle size analysis, the mean diameter (*d*) was obtained. Standard BS EN 1235 (1995) also recommends the characterisation of fertiliser materials by providing the values of percentiles $d_{16}$, $d_{50}$ and $d_{84}$. These correspond to particle diameters (mm) below which 16%, 50% and 84% (by weight) of the material respectively can be collected after sieving (Persson, 1996). The $d_{50}$ value equates to the median particle diameter (Miserque et al., 2008). Based on these percentiles, a granulometric spread index (GSI, %) was derived using Equation [1] (Lance, 1996; Miserque et al., 2008).

$$GSI = \frac{d_{84} - d_{16}}{2 \times d_{50}} \times 100 \quad \textbf{[1]}$$

The GSI was used to provide an indication of the likelihood of the materials to undergo segregation during transport, handling, loading and spreading. Segregation is the re-arrangement of particles as a result of differences in their physical characteristics; in particular, the size ratio of particles (Lance, 1996; Bridle et al., 2004). The GSI is more commonly used to characterise blended than complex and compound fertilisers; however, its application to this situation may be justified given the relatively wide range of particle sizes encountered in the samples. Based on Paré et al. (2009), a uniformity index ($U_i$, %) was calculated which is the ratio times 100 of the two extreme sizes in the range of particles retained at the 95% ($d_{95}$) and at the 10% ($d_{10}$) levels respectively (CFI, 2001). Larger $U_i$ numbers means more uniform the granulometric distribution in the sample.

Untapped bulk density ($\rho_b$; kg $m^{-3}$) was determined based on BS EN 1236 (1995) by pouring of a fertiliser sample from a funnel into a measuring cylinder of known volume and weighing of the contents and the cylinder. Particle density ($\rho_p$; kg $m^{-3}$) was determined by measuring the diameter, and calculating the volume, of individual fertiliser particles from a sample containing 100 particles and by recording the mass of the particles individually. Particle diameter was measured using a digital vernier calliper in two perpendicular directions to better account for unevenness in the particles' shape.

Subsequently, the mean of the two measured diameters was obtained which was value used to calculate the volume of the particles; these were regarded as spherical (Mennel and Reece,

1963). The porosity of untapped granules ($\eta$; $m^3\ m^{-3}$) was calculated with Equation [2] (Blake and Hartge, 1986; Paré et al., 2009):

$$\eta = 1 - \frac{\rho_b}{\rho_p} \quad \textbf{[2]}$$

Static particle strength ($\tau$; $N\ mm^{-2}$) was determined using an Instron 1122 apparatus (Instron, 1975) which consists of a frame and a moving crosshead. Particles were placed individually between the crosshead and the bottom of the frame. The crosshead (tip rod =1 $mm^2$) moved downwards at a speed of 95 mm $min^{-1}$. The device has a load-cell that senses the vertical load applied to the particle and it was connected to a laptop computer which recorded the force applied versus the time. The force required to induce the breaking of the particle (Hofstee and Huisman, 1990) or its complete compression, as determined by visual assessment, was recorded. For known crosshead speed, the vertical load versus time plot was converted to vertical load versus displacement (Gouw and Wevers, 1982). Measurements were conducted three times (n=3) for a selected range of particle diameters (from 2.85 to 6.30 mm; except for urea: from 2.85 to 4.75 mm). The total surface area per unit mass ($A$; $m^2\ kg^{-1}$) of a fertiliser sample was estimated from the particle size distribution with Equation [3] (ASABE, 2006).

$$A = \left( \frac{\beta_s W_t}{\beta_v \rho_p} \right) \exp\left[ 4.5 \times S_{\ln}^2 - \ln d_{gw} \right] \quad \textbf{[3]}$$

Where: $\beta_s$ ($\approx \pi$) and $\beta_v$ ($\approx \pi/6$) are the shape factors for calculating surface area and volume of particles respectively, $\rho_p$ is the particle density (g $cm^{-3}$), $S_{\ln}$ ($\approx 2.3\log[d_{50}/d_{16}]$) is the log-normal geometric standard deviation by mass in natural logarithm, $d_{gw}$ ($\approx d_{50}$) is the geometric mean particle diameter (cm) and $W_t$ is the mass of the sample (g).

### *Statistical analyses*

Statistical analyses were undertaken using GenStat 14th Edition. For density properties, untapped porosity and particle strength analyses involved ANOVA and least significant differences to compare the means with a probability level of 5% (LSD 5% level). For particle size distribution analyses involved t-tests (95% confidence level) to compare the mean particle size and F-tests ($\alpha$ =0.05; 100 degrees of freedom) to compare the variation in the particle size distribution between fertilisers. F-tests used a critical F-value of 1.41 (Rohlf and Sokal, 1995).

## Results

### *Chemical composition*

The results of the chemical analyses conducted on OMF and biosolids samples are summarised in Table 1.

Table 1: Chemical analyses conducted on $OMF_{15}$, $OMF_{10}$ and biosolids granules. *SD* is the standard deviation, n=12 except for heavy metals and water soluble P n=3.

| **Fertiliser material** | **$OMF_{15}$** | **$OMF_{10}$** | **Biosolids granules** |
|---|---|---|---|

| Determination | Mean | *SD* | Mean | *SD* | Mean | *SD* |
|---|---|---|---|---|---|---|
| Total N (%, w $w^{-1}$) | 13.0 | 2.75 | 9.0 | 1.55 | 4.5 | 0.73 |
| Total $P_2O_5$ (%, w $w^{-1}$) | 3.8 | 0.23 | 4.3 | 0.52 | 5.5 | 0.97 |
| Total $K_2O$ (%, w $w^{-1}$) | 3.2 | 0.92 | 3.0 | 0.61 | 0.2 | 0.07 |
| Total Cd (mg $kg^{-1}$) | 0.98 | 0.07 | 0.98 | 0.15 | 1.20 | 0.03 |
| Total Cu (mg $kg^{-1}$) | 268.4 | 10.7 | 264.2 | 11.0 | 329.3 | 11.6 |
| Total Zn (mg $kg^{-1}$) | 422.7 | 3.8 | 422.2 | 5.6 | 493.0 | 5.1 |
| Water soluble P (%, w $w^{-1}$) | <0.10 | 0.02 | 0.10 | 0.02 | 0.20 | 0.05 |
| N:$P_2O_5$ ratio | 3.4 | 0.75 | 2.1 | 0.41 | 0.85 | 0.29 |

$OMF_{10}$ and $OMF_{15}$ were not strictly made to specification. On average, the concentrations of nitrogen, phosphate and potash were slightly lower than the corresponding formulations which require optimisation of the coating technology. Figure 2 shows a sample of $OMF_{15}$ produced after coating biosolids granules with urea and potash.

Figure 2: A sample of $OMF_{15}$ with particles in the range of 1.18 to 5.60 mm in diameter.

Heavy metals (Cd, Cu and Zn) content were below the recommended limit values given in EC Sewage Sludge Directive 86/278/EEC (CEC, 1986) and therefore in compliance with the Sludge (Use in Agriculture) Regulations 1989 S.I. No.: 1263. The relatively low concentration of water soluble P suggested that the majority of the phosphorus in OMF and biosolids is not readily available for plant uptake. This responds to the removal of phosphorus during the wastewater treatment process by means of precipitation which is conducted with the use of $FeCl_3$. The phosphorus removed is subsequently incorporated into the sludge (Farrant et al., 2008).

### *Physical properties*

The results of the measured physical properties are summarised in Table 2. The particle size and size distribution of OMF and biosolids granules varied between samples due to difficulties encountered during the coating process. Loss of heat occurred during the spraying of melted

urea which meant that urea droplets solidified before being attached to the biosolids granule producing unbounded urea and dust. Due to the relatively high temperatures used in the process (range of 120 to 130°C), fine particles of urea re-melted and aggregated to biosolids granules. As a result, the thickness of the coating was not uniform for all granules which resulted in a relatively wide range of particle sizes. Particles ranged between <0.60 mm (up to 3% and 6% in $OMF_{10}$ and $OMF_{15}$ respectively) and 25 mm (up to 5% and 2% in $OMF_{10}$ and $OMF_{15}$ respectively) in diameter. The two OMF products did not show significant differences in the mean particle diameters but these were significantly higher (t-values >1.96) compared with biosolids granules and urea. The F-tests indicated significant differences (F-values >1.41) between the two OMF products compared with urea for all fertiliser samples.

Overall, there were significant differences in bulk density depending on the fertiliser type ($P<0.05$). This effect was mainly due to the value encountered for urea which was on average 20%-30% higher compared with the other fertiliser materials. The measured bulk densities of biosolids granules, $OMF_{10}$ and $OMF_{15}$ were not significantly different for an LSD value (5% level) of 110 kg $m^{-3}$. There were significant differences ($P<0.05$) in particle densities but the overall effect was due to urea which was on average 6% to 10% higher compared with the other fertiliser materials. Similarly, biosolids granules, $OMF_{10}$ and $OMF_{15}$ were not significantly different for an LSD value (5% level) of 73 kg $m^{-3}$.

Table 2: Physical properties determined on $OMF_{15}$, $OMF_{10}$, biosolids granules and urea samples. *SD* is the standard deviation.

| **Fertiliser material** | **$OMF_{15}$** | | **$OMF_{10}$** | | **Biosolids granules** | | **Urea** | | **Unit** |
|---|---|---|---|---|---|---|---|---|---|
| Physical property | Mean | *SD* | Mean | *SD* | Mean | *SD* | Mean | *SD* | - |
| $d_{16}$ | 2.52 | 1.0 | 2.40 | 0.6 | 2.74 | 3.1 | 2.52 | - | mm |
| $d_{50}$ | 4.60 | 2.8 | 4.45 | 2.8 | 4.87 | 5.6 | 3.03 | - | mm |
| $d_{84}$ | 7.24 | 4.4 | 6.91 | 5.2 | 7.08 | 7.5 | 3.73 | - | mm |
| *d* | 5.42 | 2.6 | 5.28 | 2.9 | 4.97 | 5.5 | 3.10 | 0.35 | mm |
| GSI | 49.3 | 14.3 | 43.6 | 16.1 | 48.9 | 7.7 | 20.0 | - | % |
| *A* | 4.90 | - | 5.76 | - | 4.08 | - | 1.61 | - | $m^2\ kg^{-1}$ |
| $U_i$ | 15.7 | 9.7 | 22.7 | 16.2 | 24.8 | 4.8 | 57.7 | - | % |
| $\rho_b$ | 603 | 73.1 | 623 | 85.5 | 578 | 87.2 | 748 | 5.7 | kg $m^{-3}$ |
| $\rho_p$ | 1357 | 178 | 1297 | 263 | 1333 | 315 | 1432 | 255 | kg $m^{-3}$ |
| $\eta$ | 0.55 | 0.05 | 0.52 | 0.07 | 0.56 | 0.07 | 0.49 | <0.01 | $m^3\ m^{-3}$ |
| $\tau$ (*d* =2.85 mm) | 6.31 | 0.10 | 5.19 | 0.08 | 6.41 | 0.08 | 3.73 | 0.06 | *N* $mm^{-2}$ |
| $\tau$ (*d* =3.70 mm) | 3.56 | 0.07 | 2.70 | 0.04 | 4.11 | 0.03 | 3.06 | 0.05 | *N* $mm^{-2}$ |
| $\tau$ (*d* =4.75 mm) | 2.24 | 0.03 | 2.24 | 0.04 | 1.01 | 0.01 | 1.38 | 0.02 | *N* $mm^{-2}$ |
| $\tau$ (*d* =6.30 mm) | 1.25 | 0.05 | 1.14 | 0.01 | 1.24 | 0.02 | - | - | *N* $mm^{-2}$ |

The calculated values of porosity were not significantly different ($P>0.05$) but the difference between urea and biosolids granules appears to be significant for an LSD value (5% level) of 0.08 $m^3\ m^{-3}$. The relatively lower value of porosity in urea samples was expected given that $\eta$ is calculated from bulk and particle densities (Paré et al., 2009). Allaire and Parent (2004)

encountered higher total porosity values in materials with more organic matter content which explains the relatively higher value of $\eta$ in biosolids compared with the two OMF products.

The surface area of urea was significantly lower than the two OMF and biosolids, possibly due to its relatively higher particle density. The same can be stated for comparisons between $OMF_{10}$ and $OMF_{15}$ despite that the percentage of fine particle was, on average, slightly higher in the former compared with the latter. The relatively greater surface area in the two OMF products compared with biosolids will enhance contact of particles with soil and water following spreading as observed in the study of Allaire and Parent (2004). Given the relatively low degradability of the organic-N fraction in OMF (Antille et al., 2012), increased surface area and particle contact with soil will favour the release of nutrients following soil application to a greater extent compared with biosolids. Compression tests showed that OMF and biosolids granules undergo deformation followed by multiple fractures without disintegration of the granule when vertical load was applied (Figure 3).

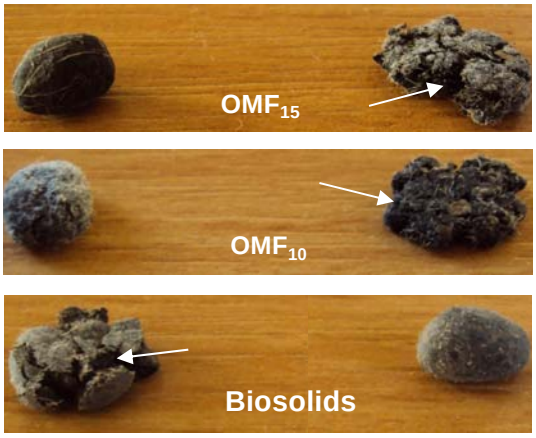


Figure 3: $OMF_{15}$, $OMF_{10}$ and biosolids granules (range of 3.35 to 4.00 mm). The arrows show the multiple failures produced during the compression tests.

Unlike urea particles, OMF and biosolids granules behaved in a plastic fashion and they did not show a characteristic force that induced the breaking of the particle. Deformation started immediately after the tip rod made contact with the granule and the test was allowed to progress until the particles were fully compressed, as recorded by visual assessment. As a result, for OMF and biosolids, the value of $\tau$ reported in Table 2 was obtained by dividing the force required to induce the full compression of the particle by its cross sectional area. Depending on the fertiliser material and the particle diameter, full compression was observed when the vertical load applied was approximately in the range of 18 to 44 *N*. This response of the materials was attributed to the moisture content (range of 10.7% to 17.3% w $w^{-1}$) and their organic nature. Figure 3 shows a vertical load *vs*. displacement diagram for particles of urea and $OMF_{10}$. It can be seen that the particle of urea breaks at 33 *N* whereas for $OMF_{10}$, the granule compresses further requiring a force of 29 *N* to reach full compression but displacement is approximately double.

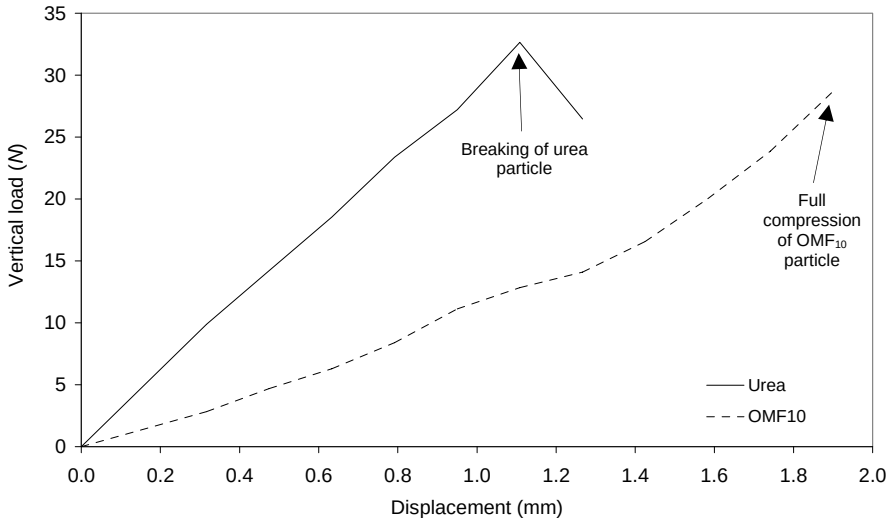


Figure 3: Vertical load *vs*. displacement recorded in particles of urea and $OMF_{10}$ (particles size range: 3.35 to 4.00 mm).

Due to the behaviour exhibited by OMF and biosolids granules, the statistical analysis to determine differences in the force required to induce the breaking of the particles was only conducted for particles of urea. Particles in the range of 3.35 to 4.00 mm in diameter required a significantly ($P<0.001$) higher force (32.9 *N*) compared with those in the range of 2.36 to 3.35 mm (23.8 *N*), and 4.00 to 5.50 mm (24.4 *N*) respectively which were not statistically different for an LSD value (5% level) of 1.26 *N*.

## Discussion

Miserque and Pirard (2004) indicated that granulometric segregation in bulk blended fertilisers can be minimised at GSI values of 15 or lower while values above 25 produce severe segregation. Segregation is not limited to blends as it takes place in complex and compound fertilisers which have a relatively wide range of particle sizes (Crowther, 1958; Lance, 1996). Segregation results in uneven distribution of nutrients in the field (Patterson and Reece, 1962) not only with blends but also with low quality compound fertilisers whose particles do not have similar chemical composition (Bridle et al., 2004). When segregation occurs before loading the fertiliser spreader, the spreading width can be affected (Bradley and Farnish, 2005). This is due to smaller and larger particles coming through in turns during loading and being subsequently delivered at different times during spreading (Bradley and Farnish, 2005). As a result, the uniformity of distribution both longitudinally and transversely can be adversely affected (Hofstee and Huisman, 1990; Persson, 1996; Bradley and Farnish, 2005).

On average, the percentage of fine particles (<1.18 mm) in the two OMF products accounted for about 4% (by weight) across all fertiliser samples (range: 0.15% to 16.5% by weight). These particles must be maintained to a minimum since the fraction below 1 mm is greatly responsible for the increase in the coefficient of variation during broadcast spreading (Kämpfe et al., 1982). The values of GSI reported by Miserque and Pirard (2004) provide a valuable threshold to compare against those obtained for OMF. Given the relatively wide range of particle sizes encountered, it can be stated that particle segregation is therefore likely to occur affecting distribution uniformity during broadcast spreading.

The compression tests demonstrated that OMF and biosolids granules did not show a characteristics force that induced the fracture of the particles. Instead, these deformed permanently when a relatively small force was applied and behaved in a plastic way. An important feature was that these materials exhibited multiple failures and they did not disintegrate into smaller particles as it was observed with urea when the breaking force was reached. Studies with urea (Hignett, 1985) showed that this force must be greater than approximately 15 *N* to avoid particle fracture during handling. At this equivalent force, $OMF_{10}$ particles had compressed to about 50% (Figure 3). Particle deformation influences the aerodynamic properties of the material as a result of changes in the particle shape. Miller (1996) indicated that particle shape is related to spreader distribution and metering flow performances as it affects particle motion in the distributor. Since one of the reasons for the relatively low particle strength was the moisture content of the material, it is suggested that this should be maintained at about 10% w $w^{-1}$. Lower values can significantly increase the cost of energy during the granulation process (Gedara, 2009). The use of plastic packaging for OMF may be recommended to prevent increases in moisture content of the material. Urea particles exhibited breaking forces which are considered to be satisfactory as they were above the lower limit suggested by Hignett (1985).

Fertilisers with a wide size range of particle sizes can suffer from caking due to the increased number of contact points and the relatively high bond strength per unit mass of the fertiliser (UNIDO/IFDC, 1998). Caking is likely to be enhanced when increased porosity and low particle strength are combined (UNIDO/IFDC, 1998) which was observed in OMF and biosolids granules. An increase in the percentage of fine particles results in increased particle-contact area which combined with low particle strength can produce compaction of the material during storage. Allaire and Parent (2004) highlighted that particle strength increases with density which agrees with the relatively higher density and strength encountered for particles of urea compared with OMF and biosolids granules. The values of porosity encountered in the two OMF products were relatively lower than those of OMF compounds reported by Paré et al. (2009) but of similar magnitude to those obtained by Allaire and Parent (2004) for bulk blended and compound organic-based fertilisers.

The mean values of bulk density for urea corresponded with those reported in the literature (UNIDO/IFDC, 1998); however, the two OMF products had relatively lower values compared with other compound organic-based fertilisers (Allaire and Parent, 2004). Fertiliser materials with low bulk density lead to increased cost of transport and spreading (Allaire and Parent, 2004). The relatively lower bulk density of biosolids compared with OMF is explained by higher organic matter in former material compared with the latter.

Studies (Hoffmeister, 1982; Nielsson, 1987) indicated that mixing of fertilisers may be restricted when differences in the size grade number between the materials are large. Therefore, due to the relatively large differences in particle size, size distribution and particle density encountered between OMF and urea, the bulk mixture of the two fertiliser types may not be recommended. Based on the relationship between flow time and bulk density encountered by Miller (1996) for straight N fertilisers, and given the differences in bulk density that exist between OMF and urea, a mixture of the two materials may result in significant differences in their flow time which will affect spreading uniformity.

Particle density for urea was within the range (from 1250 to 1500 kg $m^{-3}$) reported in the literature (Hoffmeister et al., 1964; Aphale et al., 2003; Parkin et al., 2005) which confirmed that the technique used for measuring particle density was adequate. For OMF, mean values of particle density were lower than those reported by Allaire and Parent (2004) for bulk-blended and compound organic-based fertilisers (range of 1540 to 2270 kg $m^{-3}$) but within the range (from 900 to 1580 kg $m^{-3}$) of values encountered by Paré et al. (2009).

The relatively lower particle density of OMF compared with mineral fertilisers could limit the maximum spreading width; hence, the application of the material at standard tramline spacing. Given that OMF granules did not shatter during the compression tests, it may be possible to set the disc of the fertiliser spreader at higher than the standard rotational velocity which may enable application of the material at greater spreading widths. However, because of greater forces exerted on the particles at higher rotational velocities, deformation of the granules can occur which can affect their aerodynamic properties.

Antille (2011) reported satisfactory results from distribution uniformity and machinery calibration tests conducted with OMF using a pneumatic fertiliser applicator Kuhn 2212. The pneumatic applicator performed relatively well when delivering an application rate equivalent to 455 kg $ha^{-1}$ of OMF which was uniform both across the working width of the machine and along the tramline. The data reported by Antille (2011) showed that there were no significant differences (P=0.572) in the amount of fertiliser collected in three sets of nine trays (dimensions 0.5 × 0.5 × 0.15 m) placed at 10 m intervals along the tramline. The author observed that the variation (CV =12.4%) in the amount of fertiliser collected in the trays was mainly due to fine particles (<1.18 mm) which originated from the disintegration of large aggregates of urea (>5.50 mm) in the hopper and in the boom during the fertiliser application. Compression tests conducted by Antille (2011) showed that these aggregates of urea break when a relatively small force (vertical load =4.43 *N*) is applied. Despite this, the above results demonstrated the suitability of OMF for application with pneumatic applicators but further work is required to determine whether this type of material can be uniformly applied with twin discs spreaders.

In this respect, a model developed for fertiliser particle distribution studies (Antille, 2011; Antille et al., 2013) showed that OMF particles should be between 1.10 and 5.50 mm in diameter with about 80% of the particles in the range of 2.25 to 4.40 mm to enable application with twin discs fertiliser spreaders at 18 m tramline spacing. This requires that particles leave the spinning disc with velocities in the range of 20 to 40 m $s^{-1}$ for discs set at angles between 0° and 10°, and at 1 m above the ground level. Therefore, the particle size and size distribution of the materials presented in this present study will require optimisation to enable broadcast spreading at conventional tramline widths.

## Conclusions

1. The two organomineral fertiliser products developed will ensure that the use of biosolids in agriculture is maximised in areas close to production sites. The proposed $OMF_{15}$ and $OMF_{10}$ formulations were based on relatively high $N:P_2O_5$ ratios which will minimise potential build-up of soil P in those areas; hence, a wider range of land-bank can receive organomineral fertilisers.
2. The physical properties investigated showed that field application with standard broadcast fertiliser spreading equipment may be possible to perform at 18 m tramline spacing. However, there is a need to improve the quality control for organomineral fertilisers with particular regards to particle size and size distribution which showed some variability in the samples analysed. Field spreading tests using this type of organomineral fertilisers require investigation.
3. Optimisation of the particle size and size distribution are required for safe spreading on the land to minimise adverse effects on the environment linked to nutrient losses due to poor use efficiency by crops, and loss of crop productivity. Initial efforts should be made to perfect $OMF_{15}$ as this product has higher N content and it could better fit the needs of the market in the proximity of wastewater treatment works in the NW region of England.

4. The conversion of biosolids into organomineral fertilisers addresses an important issue of nutrient cycling between urban and agricultural ecosystems. Further, the cost of disposal of sewage sludge can be significantly reduced while it enables wastewater companies meeting recycling targets. Farmers are expected to reduce the reliance on mineral fertilisers; hence, fertiliser input costs while maintaining overall soil fertility.


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